Annex A: Data related to the comparison between: US Pat. No. 5,252, 537 and US App. No. 10/551, 635

Brief Summary

As discussed below in this Annex (Annex A), colorless flowers cannot be obtained with the methods taught by U.S. Pat. No. 5,252,537 to DeWinter-Scailteur ("DeWinter-Scailteur"), whereas substantially colorless flowers may be obtained by the three-step dehydration process of the methods claimed in U.S. App. No. 10/551,635 to Silva ("the present Application").

In the DeWinter-Scailteur process, pigments remain in the solvent, staying in contact with the flowers during the dehydration step. Therefore, it is not possible to obtain colorless flowers using the process taught by DeWinter-Scailteur because flower pigments remain in the solvent rendering the flowers a yellowish color.[1]

In contrast, flowers obtained using the three-step (or more) dehydration process claimed in the present Application are ideal for a next step of infiltration. As the flowers pass through the three or more dehydrating steps, pigments are extracted in such a way that, at the end of the dehydrating steps, the flowers are substantially decolorized and are ready to receive any color in the subsequent step.

To demonstrate the technical advantages of the claimed methods of the present Application, test data showing that the use of three dehydration steps rather than one dehydration step is provided.[2] Please note that for the three-step dehydration method of the present Application, the first dehydrating step uses a mixture having more than 70% solvent, the second dehydrating step uses a mixture having more than 80% solvent, and the third dehydrating step uses a mixture having more than 90% solvent. For the

[1] See Annex A, Figure 7.
[2] See Annex B.

dehydration method taught by DeWinter-Scailteur, a mixture of more than 90% solvent was used. The data in Annex B shows that the product obtained using the three dehydrating steps as claimed in the present Application has less moisture content than the one-step dehydration method. The flower water content for the product obtained from the three dehydrating step method as claimed in the present Application is less than 1.35%, whereas the flower water content for the product obtained from the one dehydrating step is about 5.11%.[3]

The data in Annex B also shows that solvent consumption is less using the three dehydrating steps, as claimed by the present Application, than when using a one-step dehydrating process.[4] Using approximately the same amount of solvent (440 ml), only two flowers are produced using a one-step dehydration method whereas using the three-step dehydration method, five flowers are produced.[5]

A flower's whitening achieved using the three or more dehydrating steps claimed in the present Application could not be derived using the processes taught by DeWinter-Scailteur or by the processes taught in U.S. Patent No. 5,677,019 to Carstairs et al. ("Carstairs"). Carstairs teaches a method of preserving a plant's natural color (and, by definition, does not teach a method for removing color or pigment), so Annexes A and B refer to the teachings of DeWinter-Scailteur as compared to the present Application.

A brief summary of the results obtained by the experimental procedures and data disclosed in this Annex (Annex A) and in Annex B is provided in Table 1 below.

DeWinter-Scailteur	**The present Application**
Less than three dehydration steps	Three-step dehydration method
Flowers obtained have a greasy	Flowers obtained last longer and

[3] See Annex B, Graphic 2.
[4] See Annex B, Graphic 3.
[5] See Annex B, Graphic 2.

appearance and a plastic texture; flowers fail to show smoothness and durability, required by market demands (see Annex A, Figure 7)	have the appearance and texture of fresh flowers (see Annex A, Figure 8).
Not as economically favorable	More economically favorable
Failure to remove all natural pigments from flowers (see Annex A, Figure 7)	Substantially all natural pigments removed (see Annex A, Figure 8)

TABLE 1

Experimental Procedures

The experimental procedure outlined below is based on the teachings of DeWinter-Scailteur:

- Fresh flowers which are not too closed and not too opened are installed in a flower-holder grid so that they are well supported without touching each other.
- This flower-holder grid, filled with flowers to be treated, is deposited in a receptacle specially provided for this purpose, packed with a bed of molecular sieve with a porosity ranging from 3 angstroms over a thickness of 2 cm.
- To ensure the dehydration of the fresh natural flowers the mixture of organic solvents is poured onto the whole until the level exceeds the level of the flowers by about 2 cm.
- The receptacle is closed hermetically and the solvents are left to act for at least 24 hours.
- At the end of the dehydration, the grid containing the flowers is taken out and the solvents are allowed to drain out in order then to transfer the grid with the flowers to a new receptacle for the infiltration stage. The spent solvents are anhydrous and are recovered for subsequent use. The molecular sieve employed and saturated with water can be regenerated by aeration and heating.
- The receptacle for the infiltration stage also contains a layer of molecular sieve enabling a possible quantity of residual water to be trapped.
- The receptacle is then filled with a mixture of anhydrous solvents and of PEG (polyethylene glycol). These solvents promote the entry of the replacement product.
- Since PEG 1000 was not employed in the solid state, it uses pre-dissolution in anhydrous organic solvents which will be of the same kind as those employed for the dehydration, in order to promote the exchanges within the cells.
- Use is preferably made of cellosolve or monomethylene glycol monomethyl ether, mixed with acetone in proportions 70/30.
- Use 15% of PEG 400, 45% of PEG 1000 and 40% of solvents for infiltration mixture.
- The receptacle is closed hermetically to avoid evaporation. The solution is allowed to act for 24 hours.

- This is then followed by the draining and drying stage, to remove the residual part of the solvents so that the polymers resume their solid state, without spoiling or changing the anatomical structure of the petals.
- To this end, the flower-holder grid with the infiltrated flowers is deposited on a new molecular sieve, this time with a preferred porosity of 10 angstroms.
- The sieve adsorbs the water and the organic solvents of low molecular weight while supporting the petals mechanically and prevents them from distorting.
- Once dried, the flower reabsorbs a little moisture of atmospheric origin, and this increases its suppleness and its plasticity.

The experimental procedure outlined below is based on the present Application:

- Prepare 72 rose flowers for preservation:
  (1) Select and cut 72 flowers and immerse the stem of the flowers in water over a day.
  (2) Separate the non-stem portion from the stems of the flowers.
- Assemble a supporting device, the supporting device comprising four grids:
  (1) Place flowers in an opening of the grid.
  (2)Assemble the 4 grids on a central axle of the supporting device, wherein the one grid is placed at 7 cm along the central axle from an adjacent grid, distance sufficient to prevent the flowers from being crushed by an adjacent grid.
- Perform a first dehydrating step, the first dehydrating step comprising:
  (1) Filling the supporting device with flowers.
  (2) Placing the supporting device into a reactor.
  (3) Filling the reactor with a first mixture that comprises 24 liters of anhydrous ethanol and 8 liters of water, passed from a feeder tank, maintaining it at a temperature of 80 °C. Introduce air, if desired, to maintain 10 psi manometric pressure and avoid boiling.
  (4) Maintaining the flowers into the first mixture at 80 °C temperature of and 10 psi pressure for 60 minutes.
  (5) Extracting the first mixture from the reactor.
- Perform a second dehydrating step, the second dehydrating step comprising:
  (1) Filling the reactor with a second mixture that comprises 27.2 liters of anhydrous ethanol and 4.8 liters of water, passed from a feeder tank, maintaining it at a temperature of 65°C. Introducing air, if desired, to maintain 10 psi manometric pressure and avoid that mixture boil.
  (2) Maintaining the flowers into the second mixture at 65 °C temperature and 10 psi pressure for 60 minutes;
  (3) Extracting the second mixture from the reactor.
- Performing a third dehydrating step, the third dehydrating step comprising:
  (1) Filling the reactor with a third mixture that comprises 30.4 liters of anhydrous ethanol and 1.6 liters of water, passed from a feeder tank, maintaining it at a temperature of 65oC. Introduce air, if desired, to maintain 10 psi manometric pressure and avoid boiling.
  (2) Maintaining the flowers into the third mixture at 65 °C temperature and 10 psi pressure for 60 minutes.

(3) Extracting the third mixture from the reactor.

- Perform an infiltration step, the infiltration step comprising:
  (1) Immersing the flowers in a bath mixture that comprises a blend of 23.4 liters of anhydrous ethanol, 6.4 liters of PEG-400 and 2.2 liters of water passing it from a feeder tank, maintaining it at a temperature of 65 °C. Introduce air, if desired, to maintain 10 psi manometric pressure to avoid boiling.
  (2) Maintaining the flowers into the bath mixture at 65 °C temperature and 10 psi pressure for 8 hours.
  (3) Extracting the bath mixture from the reactor.
  (4) Setting the reactor to be under vacuum between 7 psi and 10 psi during one hour. Then, interrupting the vacuum, opening the reactor, and taking out the supporting device and grids together with the flowers.
- Performing an evaporation step, the evaporation step comprising the bath mixture being substantially removed from the flowers and the fourth mixture been substantially evaporated applying an evaporating temperature. Maintaining the flowers at 65 °C temperature for 2 hours.